

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

| | | | |
|--|--|---|---|
| (51) International Patent Classification ⁶ : C09J 7/02, C08L 23/02, C09J 123/02 // (C08L 23/02, 23:06, 23:12) | | A1 | (11) International Publication Number: WO 97/05206 |
| | | | (43) International Publication Date: 13 February 1997 (13.02.97) |
| (21) International Application Number: PCT/GB96/01822 (22) International Filing Date: 29 July 1996 (29.07.96) (30) Priority Data: 9515827.5 2 August 1995 (02.08.95) GB (71) Applicant (for all designated States except US): SCAPA GROUP PLC [GB/GB]; Oakfield House, 93 Preston New Road, Blackburn, Lancashire BB2 6AY (GB). (72) Inventors; and (75) Inventors/Applicants (for US only): RICHARDSON, Peter, James [GB/GB]; 132 Moor Lane, Woodford, Cheshire SH7 1PJ (GB). SALTHOUSE, Peter, William [GB/GB]; 54 Glebelands Road, Knutsford, Cheshire WA16 9DZ (GB). PRESTON, Joseph [GB/GB]; 39 Cloverfields, Haslington, Crewe, Cheshire CW1 1AL (GB). STEWART, Edward, John [GB/GB]; 47 Sandywell Street, Higher Openshaw, Manchester M11 1BD (GB). (74) Agents: GOODWIN, Mark et al.; Wilson Gunn M'Caw, 41-51 Royal Exchange, Cross Street, Manchester M2 7BD (GB). | | (81) Designated States: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i> | |
| (54) Title: PRESSURE SENSITIVE ADHESIVE TAPE | | | |
| (57) Abstract A pressure sensitive adhesive tape comprises a tape body coated on one side thereof with an adhesive. The tape body comprises the following components: (a) from 40 % to 85 % wt. of at least one copolymer of olefin with vinyl- or acrylic esters; (b) from 0 % to 20 % wt. of low density (less than 0.9 g cm ⁻³) polyethylene; (c) from 20 % to 55 % wt. of inorganic filler and/or flame retardant consisting of any of the following either alone or in combination: aluminum trihydrate filler, zinc borate, an organic polyphosphate, polyammonium phosphate or magnesium hydroxide; and (d) from 0 % to 5 % wt. of a silane coupling agent. | | | |

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

| | | | | | |
|----|--------------------------|----|---------------------------------------|----|--------------------------|
| AM | Armenia | GB | United Kingdom | MW | Malawi |
| AT | Austria | GE | Georgia | MX | Mexico |
| AU | Australia | GN | Guinea | NE | Niger |
| BB | Barbados | GR | Greece | NL | Netherlands |
| BE | Belgium | HU | Hungary | NO | Norway |
| BF | Burkina Faso | IE | Ireland | NZ | New Zealand |
| BG | Bulgaria | IT | Italy | PL | Poland |
| BJ | Benin | JP | Japan | PT | Portugal |
| BR | Brazil | KE | Kenya | RO | Romania |
| BY | Belarus | KG | Kyrgyzstan | RU | Russian Federation |
| CA | Canada | KP | Democratic People's Republic of Korea | SD | Sudan |
| CF | Central African Republic | KR | Republic of Korea | SE | Sweden |
| CG | Congo | KZ | Kazakhstan | SG | Singapore |
| CH | Switzerland | LI | Liechtenstein | SI | Slovenia |
| CI | Côte d'Ivoire | LK | Sri Lanka | SK | Slovakia |
| CM | Cameroon | LR | Liberia | SN | Senegal |
| CN | China | LT | Lithuania | SZ | Swaziland |
| CS | Czechoslovakia | LU | Luxembourg | TD | Chad |
| CZ | Czech Republic | LV | Larvia | TG | Togo |
| DE | Germany | MC | Monaco | TJ | Tajikistan |
| DK | Denmark | MD | Republic of Moldova | TT | Trinidad and Tobago |
| EE | Estonia | MG | Madagascar | UA | Ukraine |
| ES | Spain | ML | Mali | UG | Uganda |
| FI | Finland | MN | Mongolia | US | United States of America |
| FR | France | MR | Mauritania | UZ | Uzbekistan |
| GA | Gabon | | | VN | Viet Nam |

PRESSURE SENSITIVE ADHESIVE TAPE

The present invention relates to a pressure sensitive adhesive tape.

It is conventional to bind wires and cables together using adhesive tape made from a polymeric material. The constituent polymers of such tapes are generally halogenated in order to provide the necessary flame retardance. For example, many tapes are made from polyvinyl chloride (PVC). The main problem of PVC and other halogenated polymers is that upon combustion a large volume of toxic, acidic and highly corrosive hydrogen halide smoke is liberated. In addition there has recently been much debate surrounding the environmental damage caused during the manufacture and disposal of PVC products. Hence there is a need for a halogen-free, recyclable adhesive tape. Ideally the adhesive tape should be suitable for a wide range of automotive and industrial applications and should exhibit sufficient flexibility, softness, flame retardancy and have acceptable unwind and handling properties. Most non-PVC adhesive tapes have no flame retardant properties. Those that do tend to be inflexible. The flexibility of the tape is now a crucial consideration as a common use for such tapes is to bind together the wires and cables of vehicle wiring harnesses. As these harnesses increase in complexity they include increasing numbers of wires which must be received through small holes in the vehicle body.

Thus the flexibility of the harness during installation and in service should not be unduly restricted by the adhesive

tape. Such flexibility is frequently achieved by the use of plasticisers having a relatively low molecular weight with respect to that of the host polymer. Certain of these plasticisers have been shown to be carcinogenic. Furthermore, plasticisers in general present problems due to their tendency to migrate from the film into the adhesive, resulting in changes in adhesive performance.

The present invention seeks to provide a recyclable, flexible, pressure sensitive adhesive tape having no halogen component and not being prone to the problems caused by plasticiser migration, but which has acceptable flame retardant properties.

According to the present invention there is provided a pressure sensitive adhesive tape comprising a tape body coated on one side thereof with an adhesive, the tape body comprising the following components:-

- (a) from 40% to 85% wt. of at least one copolymer of olefin with vinyl- or acrylic esters (hereinafter called "copolymer a");
- (b) from 0% to 20% wt. of low density (less than 0.9g cm^{-3}) polyethylene;
- (c) from 20% to 55% wt. of inorganic filler and/or flame retardant consisting of any of the following either alone or in combination: aluminum trihydrate filler, zinc borate, an organic polyphosphate, polyammonium phosphate or magnesium hydroxide; and
- (d) from 0% to 5% wt. of a silane coupling agent.

The tapes of the invention are sufficiently flexible and

soft for most applications and have good unwinding and handling properties such that the tapes are suitable for both automatic and manual wrapping. Sufficient flame retardancy is also exhibited to satisfy the standard tests UL510 and ISO 3795, even though the tapes do not comprise any halogenated components.

The "copolymer a" component of the tape may comprise any of the following either alone or in combination: ethylene vinyl acetate copolymer (EVA), ethylene-methyl acetate (EMA) or ethylene-butyl acetate (EBA). The preferred component is EVA.

The "copolymer a" component of the tape renders the tape with its good flexibility. The "copolymer a" preferably has a low melt flow index (MFI), i.e. an MFI value of less than 6. This reduces the tendency for stress cracks to occur in the tape, especially at higher temperatures. The "copolymer a" preferably comprises at least two vinyl acetate copolymers having different melt flow indexes. In a particularly preferred embodiment of the invention the first of said copolymers has an MFI of between 4 and 7, and ideally 6, and the second has an MFI of from 0.5 to 1, and ideally 0.75, the two copolymers being present in a ratio having a range from 3 to 1.5:1 and ideally being 2:1 (first polymer:second polymer).

The low density polyethylene is more compatible with the filler than the "copolymer a" and ensures a more homogeneous distribution of the filler.

The fillers and/or flame retardants have a low surface area so as to enhance flexibility. Fillers having a BET value

of substantially 4 are preferred.

The composition may further optionally comprise from 5% to 45% wt. of the plastomer polypropylene. Polypropylene is added in order to raise the temperature rating of the tape.

The film may be manufactured by any conventional technique, for example melt blowing. It is noted that PVC cannot be melt blown and therefore must be cast or calandered. Thus the tape of the invention is less expensive to manufacture.

The adhesive of the tape may comprise any standard adhesive for such tapes including, for example, solvent or water based or powder coated acrylic, polyurethane or polyester adhesives or natural rubber based adhesives. Heat curable adhesives are preferred.

The tapes are free of halogenated materials such as PVC. The tapes are also preferably free of plasticisers, which are prone to migration and have been shown to be carcinogenic.

The tapes are also resistant to conditions which are conventionally found in motor vehicle engines, such wires being subjected to high temperatures and contact with water and fluids such as petrol, diesel, oil, salt solution and anti freeze.

The tapes of the present invention have a temperature rating of between 105° and 130°.

In order that the present invention may be more readily understood specific embodiments thereof will now be described by way of example only with reference to the examples hereafter in which the amounts given are in php (parts per

hundred parts of polymer).

Compound A

9.59 Evatane 2805¹, 8.13 VLDPE², 82.28 Evatane 1005 VN4³, 74.26 aluminum trihydrate (BET = 4), 1.27 Irganox 1010⁴, 0.71 AMEO-T⁵, 0.60 stearic acid and 0.60 paraffin wax.

Compound B

6.2 Evatane 2805, 5.2 VLDPE, 88.60 Evatane 1005 VN4, 48.00 aluminum trihydrate (BET = 4), 1.25 Irganox 1010, 0.40 stearic acid, 0.40 paraffin wax, 0.50 AMEO-T and 3.75 black masterbatch pigment.

Compound C

5.2 Evatane 2805, 4.4 VLDPE, 90.4 Evatane 1005 VN4, 3.6 Irganox 1010, 0.2 AMEO-T, 60.00 aluminum trihydrate (BET = 7), 0.32 stearic acid and 0.32 paraffin wax.

Compound D

45 Evatane 1005 VN4, 25 Lotryl 29 MA 03⁶, 15 Lotader 3210⁷, 15 VLDPE, 1.50 Irganox 1010, 1.50 stearic acid, 1.50 paraffin wax, 90 aluminium trihydrate (BET = 7) and 2.0 black masterbatch. Tapes prepared from Compound D have a temperature rating of 125°C.

REFERENCES

- | | | |
|---|--------------|---|
| 1 | Evatane 2805 | Elf Atochem tradename for a copolymer of ethylene and vinyl acetate. Vinyl acetate content is 28% and its melt flow index is 5.0g/10 minutes. |
| 2 | VLDPE | Very low density (0.911g/cm ³) polyethylene with a melt flow index of 0.7g/10 minutes. |

6

- | | | |
|---|------------------|---|
| 3 | Evatane 1005 VN4 | Elf Atochem tradename for a copolymer of ethylene and vinyl acetate. Vinyl acetate content is 14% and its melt flow index is 0.7g/10 minutes. |
| 4 | Irganox 1010 | Ciba Geigy tradename for a standard anti-oxidant system. |
| 5 | AMEO-T | Huls AG tradename for an octyl triethoxy silane coupling agent used to coat the flame retardant (aluminum trihydrate) particles. |
| 6 | Lotryl 29 MA 03 | Elf Atochem tradename for a copolymer of ethylene and methyl acrylate. Methyl acrylate content is 29% and its melt flow index is 3.0g/10 minutes. |
| 7 | Lotader 3210 | Elf Atochem tradename for a terpolymer of ethylene, butyl acrylate and maleic anhydride. Butyl acrylate content is 9% and its melt flow index is 5.0g/10 minutes. |

A zero halogen content harness tape prepared by melt blowing compound B above had the following properties:-

- 0.12 - 0.15 mm thickness (test method AFERA 4006)
- min. 20N/cm tensile strength (test method AFERA 4004)
- min. 300% elongation at break (test method AFERA 4005)

- min. 1.5N/cm adhesion strength to steel (test method AFERA 4001)
- min. 1.0N/cm adhesion strength to itself (test method AFERA 4001)
- min 2.0N/cm unwind strength (test method AFERA 4013)
- passes the UL510 test and conforms with ISO 3795 in respect of flammability
- temperature rating of 105°C (3000 hours at 105°C).

This tape is soft and conformable, tough, easy to unwind, fire retardant, recyclable, suitable for both hand and machine applications and resistant to moisture, weather and many chemicals (including dilute acids and alkalis).

The AFERA, UL510 AND ISO 3795 test methods are well known in the automotive tape industries.

The flexibility of the tapes of the invention can be compared with that of a standard PVC tape by measuring the modulus at 35% elongation - a 25mm wide strip of a typical tape in accordance with the invention gives a value of 7 MPa, whereas a PVC strip of the same dimensions has a value of 12.6 MPa. Thus it can be shown that the modulus of the tapes of the invention is nearly 50% that of PVC; i.e. it is much more flexible.

It is to be understood that the above described embodiments of the invention are by way of illustration only. Many modifications and variations are possible within the scope of the invention.

CLAIMS

1. A pressure sensitive adhesive tape comprising a tape body coated on one side thereof with an adhesive, the tape body comprising the following components:
 - (a) from 40% to 85% wt. of at least one copolymer of olefin with vinyl- or acrylic esters (hereinafter called "copolymer a");
 - (b) from 0% to 20% wt. of low density (less than 0.9g cm^{-3}) polyethylene;
 - (c) from 20% to 55% wt. of inorganic filler and/or flame retardant consisting of any of the following either alone or in combination: aluminum trihydrate filler, zinc borate, an organic polyphosphate, polyammonium phosphate or magnesium hydroxide; and
 - (d) from 0% to 5% wt. of a silane coupling agent.
2. A tape as claimed in claim 1, wherein the said "copolymer a" comprises any of the following either alone or in combination: ethylene vinyl acetate copolymer (EVA), ethylene-methyl acetate (EMA) or ethylene-butyl acetate (EBA).
3. A tape as claimed in claim 1 or claim 2, wherein the said "copolymer a" has a melt flow index value of less than 6.
4. A tape as claimed in any preceding claim, wherein the said "copolymer a" comprises at least two vinyl acetate copolymers having different melt flow indexes.
5. A tape as claimed in claim 4, wherein a first of said at least two vinyl acetate copolymers has a melt flow index of between 4 and 7 and a second of said at least two vinyl acetate copolymers has a melt flow index in the range from 0.5

to 1.

6. A tape as claimed in claim 4 or claim 5, wherein the said at least two copolymers are present in a ratio having a range from 3 to 1.5:1.

7. A tape as claimed in any preceding claim, wherein the said inorganic filler material has a BET value of substantially 4.

8. A tape as claimed in any preceding claim, wherein the tape further comprises from 5% to 45% of polypropylene.

9. A tape as claimed in any preceding claim, wherein the present invention has a temperature rating in the range from 105°C to 130°C.

INTERNATIONAL SEARCH REPORT

International Application No

PC 1/GB 96/01822

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C09J7/02 C08L23/02 C09J123/02 //(C08L23/02,23:06,23:12)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C09J C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|----------|--|-----------------------|
| A | EP,A,0 235 930 (MINNESOTA MINNING AND MANUFACTURING COMPANY) 9 September 1987 see claims 1,2,4,5,12,14 --- | 1-9 |
| A | EP,A,0 255 866 (BEIERSDORF AKTIENGESELLSCHAFT) 17 February 1988 * Comparative examples 7,8 * --- | 1-9 |
| A | FR,A,2 242 450 (NITTO ELECTRIC INDUSTRIAL CO., LTD.) 28 March 1975 see claims 1,4 --- | 1-9 |
| A | BE,A,836 750 (ETAFIN CORPORATION S.A.) 16 April 1976 see claims 1,4,5 --- | 1-9 |
| | --- -/-- | |

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

Special categories of cited documents:

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *†* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- *&* document member of the same patent family

Date of the actual completion of the international search

29 October 1996

Date of mailing of the international search report

- 8. 11. 96

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl,
Fax (+ 31-70) 340-3016

Authorized officer

Glanddier, A

INTERNATIONAL SEARCH REPORT

International Application No
PC/GB 96/01822

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

| Category * | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|------------|---|-----------------------|
| A | <p>PATENT ABSTRACTS OF JAPAN vol. 16, no. 63 (C-911), 20 November 1991 & JP,A,03 259938 (SUMITOMO ELECTRIC IND LTD) see abstract</p> <p>-----</p> | 1-9 |

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PLI/GB 96/01822

| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
|---|---------------------|---|--|
| EP-A-235930 | 09-09-87 | US-A- 4769283 AU-B- 598214 AU-A- 6642986 CA-A- 1317819 DE-A- 3785866 DE-T- 3785866 JP-A- 62184080 KR-B- 9508188 US-A- 4904253 | 06-09-88 21-06-90 06-08-87 18-05-93 24-06-93 11-11-93 12-08-87 26-07-95 27-02-90 |
| EP-A-255866 | 17-02-88 | DE-A- 3624921 JP-A- 63035678 | 28-01-88 16-02-88 |
| FR-A-2242450 | 28-03-75 | JP-C- 950829 JP-A- 50052142 JP-B- 53028058 BE-A- 819210 DE-A- 2440286 GB-A- 1447837 US-A- 4137362 | 27-04-79 09-05-75 11-08-78 16-12-74 06-03-75 02-09-76 30-01-79 |
| BE-A-836750 | 16-04-76 | NONE | |